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# **MBE System for Antimonide Based Semiconductor Lasers**

## **Final Report**

With the approval of the program manager, the equipment purchased on this grant was changed to a Plasma Therm, Inc. SLR-770 inductively coupled plasma (ICP) processing system. This change was precipitated by two events: 1) The lack of an affordable MBE system at the amount provided by the grant and 2) the indefinite loan of a dual chamber MBE system to the University of New Mexico by Sandia National Laboratories beginning two weeks before this AFOSR DURIP grant started.

The SLR-770 has been invaluable in the study of plasma etching of AlGaAsSb and GaSb—materials that form the backbone of antimonide-based semiconductor lasers. AlGaAsSb is especially difficult to pattern with wet chemicals because it is prone to oxidation. The combination of sputtering and chemical etching in plasmas represents the only reproducible means of etching this quaternary alloy. Specifically, we have studied etch rate and selectivity of GaSb and AlGaAsSb in plasmas containing  $\text{BCl}_3$ ,  $\text{Cl}_2$ , and Ar as a function of process pressure, DC bias, ICP power, and reactant concentration. Smooth and anisotropic etching was obtained over a variety of process conditions. This research will benefit future efforts to fabricate ridge-waveguide, DFB, and etched mirror laser diodes designed with antimonide semiconductors.

Two publications have directly resulted from our work with the ICP plasma etcher. One was a presentation at the Fall 1998 Materials Research Society meeting, and the other is a paper soon to be published in the Journal of Vacuum Science and Technology.

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# III-V Antimonide Etching Characteristics in Inductively Coupled $\text{BCl}_3/\text{Cl}_2/\text{Ar}$ Plasmas

L. Zhang, L. F. Lester,  
*University of New Mexico, Center for High Technology Materials, Albuquerque, NM 87106*  
R. J. Shul, C. G. Willison,  
*Sandia National Laboratories, Albuquerque, NM 87185-0603*  
J. Hong, S. J. Pearton,  
*University of Florida, Department of Materials Science and Engineering, Gainesville, FL 32611*  
R. P. Leavitt  
*Army Research Laboratory, Adelphi, MD 20783-1145*

## Abstract

Inductively coupled plasma (ICP) etching of GaSb,  $\text{Al}_{0.9}\text{Ga}_{0.1}\text{As}_{0.07}\text{Sb}_{0.93}$  and InSb has been investigated in  $\text{BCl}_3/\text{Ar}$ ,  $\text{Cl}_2/\text{Ar}$  and  $\text{BCl}_3/\text{Cl}_2/\text{Ar}$  discharges. Etch rates and selectivities are reported as a function of ICP power, rf self-bias, chamber pressure, and gas composition. It is found that the etching of GaSb in  $\text{Cl}_2/\text{Ar}$  plasma is dominated by the chemical reaction. GaSb etch rates in excess of 2  $\mu\text{m}/\text{min}$  are achieved in  $\text{Cl}_2/\text{Ar}$  with smooth surfaces and anisotropic profiles. However, the surface of AlGaAsSb etched in  $\text{Cl}_2/\text{Ar}$  is fairly rough. In  $\text{BCl}_3/\text{Ar}$  plasmas, physical sputtering desorption of the etching products plays a dominant role. Maximum etching rates of 5100  $\text{\AA}/\text{min}$  and 4200  $\text{\AA}/\text{min}$  are obtained for GaSb and AlGaAsSb, respectively, which are much lower than those obtained in  $\text{Cl}_2/\text{Ar}$ . The surfaces of both GaSb and AlGaAsSb etched in  $\text{BCl}_3/\text{Ar}$  plasma remain smooth and stoichiometric over the entire range of plasma conditions that are investigated, suggesting that  $\text{BCl}_3$  helps to remove the Al in AlGaAsSb and produces smooth etched surfaces. By choosing  $\text{BCl}_3/\text{Cl}_2/\text{Ar}$  chemistries, etch rates in excess of 1  $\mu\text{m}/\text{min}$  for GaSb and AlGaAsSb and 0.8  $\mu\text{m}/\text{min}$  for InSb are achieved. The GaSb:AlGaAsSb etch selectivity is close to unity for a wide range of plasma conditions, which is desirable for fabricating gratings, ridge waveguides, and etched mirrors for GaSb/AlGaAsSb mid-IR laser diodes.

## Introduction

III-V antimonides have been attracting considerable interest for making mid-IR laser diodes and low power-consumption, high-speed electronics primarily due to their narrow bandgap energy, and high electron mobility and velocity. Broad area laser diodes emitting from 1.6–4.5  $\mu\text{m}^{1-3}$  as well as AlSb/InAs high electron mobility transistors (HEMTs)<sup>4</sup> have been demonstrated. Fabrication of most device structures requires well-controlled etch techniques to achieve reliable pattern transfer. Compared to wet chemical etching, dry etching provides a more anisotropic profile, greater repeatability, improved etch depth control, and higher aspect ratio. Dry etching is especially important for patterning III-Sb's with high Al concentration because wet etching of these materials yields slow etch rates and rough surfaces.<sup>5</sup> Photonic devices usually requires fast, anisotropic, and non-selective etches to form smooth and vertical sidewalls for waveguides and laser mirrors, while in case of HEMTs, high selectivity is important for accurately stopping the etch on a specific layer. Unfortunately, the energetic ion bombardment essential to achieve fast and anisotropic etching usually minimizes the etch selectivity. However, by reducing the ion energy or by increasing the chemical activity in the plasma, etch selectivity can be improved. Pearton, *et al.*, have investigated electron cyclotron resonance (ECR) etching and reactive ion etching (RIE) of InSb and GaSb using various chemistries.<sup>6-9</sup> GaSb etch rates of 5000  $\text{\AA}/\text{min}$ , 3000  $\text{\AA}/\text{min}$ , and 2500  $\text{\AA}/\text{min}$  were reported in  $\text{Cl}_2/\text{Ar}$ ,  $\text{BCl}_3/\text{Ar}$ , and  $\text{CH}_4/\text{H}_2/\text{Ar}$  ECR discharges, respectively. This paper presents results of inductively coupled plasma (ICP) etching as an attractive alternative to ECR techniques for patterning antimonides.

ICP plasmas are formed in a dielectric vessel encircled by an inductive coil into which rf power is applied. In an inductively coupled source, the plasma acts as a single-turn, lossy conductor that is coupled to a multi-turn nonresonant rf coil across the dielectric discharge chamber. The rf power is transferred from the coil to the plasma by transformer action.<sup>10</sup> Anisotropic etch profiles are obtained by superimposing an rf-induced DC self-bias on the sample to control the ion energy

independent of the ion density. Compared to ECR sources, it is believed that ICP sources are easier to scale-up, more economical in terms of cost and power requirements, and have more mature automatic tuning technology.<sup>11</sup> In this paper, ICP etch results of GaSb, AlGaAsSb, and InSb in  $\text{BCl}_3/\text{Cl}_2/\text{Ar}$  plasmas are presented. The etch rate and selectivity are reported as a function of plasma chemistry and DC self-bias.

## Experiment

The samples used in this study are undoped bulk GaSb, InSb, and  $\text{Al}_{0.9}\text{Ga}_{0.1}\text{As}_{0.07}\text{Sb}_{0.93}$  grown lattice-matched to GaSb by molecular beam epitaxy (MBE). The AlGaAsSb is capped with 100 Å GaSb layer to avoid oxidation. Etching is performed in a load-locked Plasma-Therm SLR 770 ICP reactor with the ICP source operating at 2 MHz. Energetic ion bombardment is provided by superimposing a DC self-bias that is generated by a separate 13.56 MHz source on the sample. For all experiments, the automatic matching network is set to the DC self-bias control mode that varies the input rf power to match the set point of the DC self-bias. Etch gases are introduced through an annular region at the top of the chamber. All samples are mounted using vacuum grease on an anodized Al carrier that is clamped to the cathode and backside-cooled with He gas. The temperature of the electrode is set to 10 °C. Samples are patterned with AZ 4330 photoresist. The etch depth is measured using an Alpha-step stylus profilometer after the photoresist is removed. Surface morphology, anisotropy, and sidewall undercutting are evaluated with a scanning electron microscope (SEM). Unless otherwise mentioned, the baseline etch parameters are 750 W ICP power, 100 V DC self-bias, 2 mTorr chamber pressure, 25 sccm  $\text{BCl}_3$  or  $\text{Cl}_2$  flow rate, and 5 sccm Ar flow rate.

## Results

Fig. 1 compares ICP etching of GaSb and AlGaAsSb as a function of DC self-bias in  $\text{BCl}_3/\text{Ar}$  and  $\text{Cl}_2/\text{Ar}$  discharges. Due to the surface roughness of AlGaAsSb etched in  $\text{Cl}_2/\text{Ar}$  plasma, an accurate measurement of etch depth on these samples is unachievable; therefore, only GaSb etch rates in  $\text{Cl}_2/\text{Ar}$  plasma are available. In  $\text{BCl}_3/\text{Ar}$  plasma, etch rates increase near linearly with increasing DC bias voltage, indicating physical sputtering desorption plays a significant role in  $\text{BCl}_3/\text{Ar}$  ICP etch of these materials. Etch rates of GaSb and AlGaAsSb rise by a factor of 2.5 as DC bias increases from 50 to 300 V. This result is attributed to improved bond breaking efficiency and enhanced sputtering desorption of etch products due to higher ion energies at higher DC bias. In  $\text{Cl}_2/\text{Ar}$  plasmas, the GaSb etch rate is much higher than in a  $\text{BCl}_3/\text{Ar}$  mixtures for similar process conditions, possibly due to a much higher concentration of reactive Cl in a  $\text{Cl}_2$  plasma as compared to a  $\text{BCl}_3$  plasma. Despite a significant change in DC bias from 50 to 200 V, the GaSb etch rate in  $\text{Cl}_2/\text{Ar}$  increases only ~20%. As the DC bias is increased further to 300 V, the etch rate actually decreases slightly. This weak dependence of etch rate on DC bias indicates that the

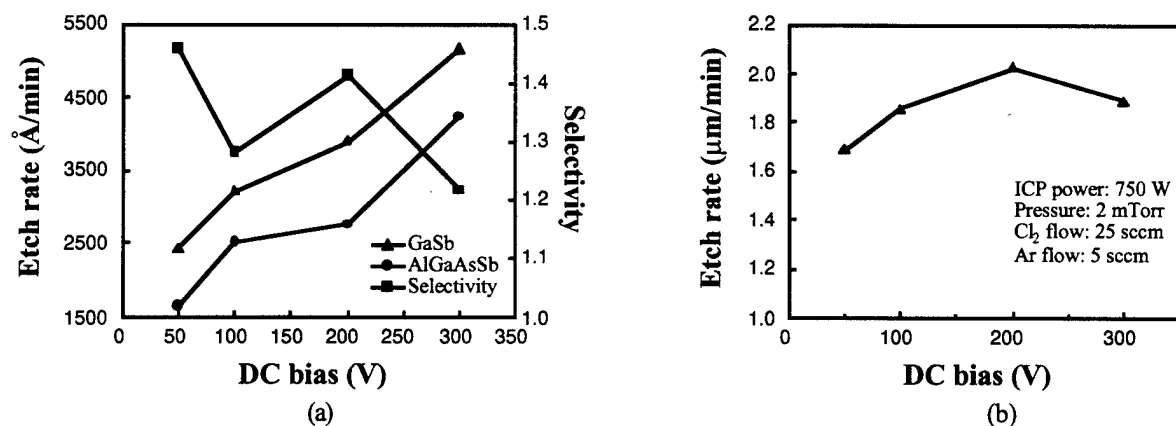


Fig. 1: ICP etch rates and selectivity of GaSb and AlGaAsSb as a function of DC bias in (a)  $\text{BCl}_3/\text{Ar}$ , and (b)  $\text{Cl}_2/\text{Ar}$  plasmas. The plasma conditions are: 750 W ICP power, 2 mTorr pressure, 25 sccm  $\text{BCl}_3$  or  $\text{Cl}_2$  flow rate, and 5 sccm Ar flow rate.

physical sputtering is not a dominant factor in  $\text{Cl}_2/\text{Ar}$  ICP etching. The decrease in etch rate at 300 V may be related to sputtering desorption of reactive species before they have time to react with the III-V antimonides at the surface. The simultaneous increase in the GaSb and AlGaAsSb etch rates in  $\text{BCl}_3/\text{Ar}$  plasma yields a relatively constant GaSb:AlGaAsSb etch selectivity of approximately  $1.34 \pm 0.12$ .

In Fig. 2 (a), etch rates and the selectivity of GaSb and AlGaAsSb are plotted as a function of percent composition of  $\text{BCl}_3$  in a  $\text{BCl}_3/\text{Ar}$  plasma. The total flow rate of  $\text{BCl}_3$  and Ar is held constant at 30 sccm. The data shows a relatively small variation in the GaSb etch rate over the entire range of  $\text{BCl}_3$  composition. This observation suggests that in a  $\text{BCl}_3/\text{Ar}$  ICP mixture, the effect of a higher concentration of reactive Cl on the GaSb etch rate (at a higher  $\text{BCl}_3/\text{Ar}$  ratio) is compensated by the effect of a lower Ar ion sputtering rate. To further verify this assumption, an ICP etch of GaSb and AlGaAsSb in  $\text{BCl}_3/\text{N}_2/\text{Ar}$  is carried out to decouple the physical and chemical component of the etching mechanism. Generally, adding  $\text{N}_2$  to a  $\text{BCl}_3/\text{Ar}$  plasma enhances the dissociation of  $\text{BCl}_3$ , resulting in a higher concentration of reactive Cl and, consequently, a higher etch rate.<sup>12,13</sup> Based on a previous study of GaN ICP etching in  $\text{BCl}_3/\text{N}_2$ , which shows a peak GaN etch rate at 40%  $\text{N}_2$ ,<sup>14</sup> a chemistry of 15 sccm  $\text{BCl}_3$ /10 sccm  $\text{N}_2$ /5 sccm Ar was chosen to etch GaSb. An etch rate of 5536 Å/min is obtained for GaSb and 4428 Å/min for AlGaAsSb, much higher than the rates obtained in the 25 sccm  $\text{BCl}_3$ /5 sccm Ar ICP discharge shown in Fig. 2(a). This outcome indicates that the etching is limited by the reactive Cl supply. Knowing that  $\text{Ar}^+$  sputters more efficiently than any ion from dissociated  $\text{BCl}_3$ , these findings confirm that the physical and chemical component are balanced in the  $\text{BCl}_3/\text{Ar}$  ICP etch, producing a relatively small variation in the GaSb etch rate with changes in  $\text{BCl}_3$  composition. In contrast, the AlGaAsSb ICP etch rate shows a significant increase when  $\text{BCl}_3$  is first introduced into the pure Ar plasma. The etch rate grows by a factor of more than 5 when the  $\text{BCl}_3$  concentration is raised from 0 to 33%, indicating that  $\text{BCl}_3$  removes Al more efficiently than Ar due to the high volatility of the  $\text{AlCl}_3$  etch product. A further increase in the  $\text{BCl}_3$  concentration shows little effect on the etch rate of AlGaAsSb. The etch selectivity between GaSb and AlGaAsSb is  $\sim 6:1$  for pure Ar plasma and drops to  $\sim 1.2:1$  as  $\text{BCl}_3$  is introduced into the plasma.

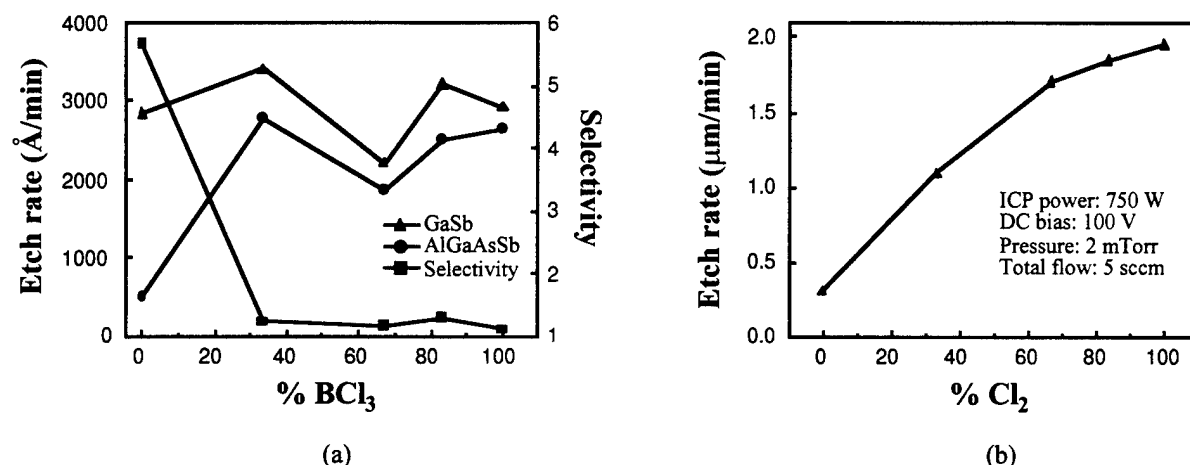


Fig. 2: (a) ICP etch rates and selectivity of GaSb and AlGaAsSb as a function of % $\text{BCl}_3$  in a  $\text{BCl}_3/\text{Ar}$  plasma, and (b) etch rate of GaSb as a function of % $\text{Cl}_2$  in a  $\text{Cl}_2/\text{Ar}$  plasma. The plasma conditions are: 750 W ICP power, 100 V DC bias, and 2 mTorr pressure. The total gas flow rate is 30 sccm.

In contrast to the  $\text{BCl}_3/\text{Ar}$  ICP etch of III-V antimonides which shows physical sputtering dominated etch characteristics due to a limited reactive Cl supply, the  $\text{Cl}_2/\text{Ar}$  ICP etch mechanism is strongly dominated by chemical reaction. Figure 2(b) shows the etch rate as a function of % $\text{Cl}_2$  in a  $\text{Cl}_2/\text{Ar}$  chemistry. The etch rate increases monotonically with increasing  $\text{Cl}_2$  percentage as would be expected from the increased reactive chlorine density in the chamber. Notice that the GaSb etch rate is substantially enhanced (by a factor of  $> 6$ ) as the  $\text{Cl}_2$  percentage increases from 0 to 100%, indicating that the chemical reaction dominates the ICP etching of GaSb in the  $\text{Cl}_2/\text{Ar}$  chemistry.

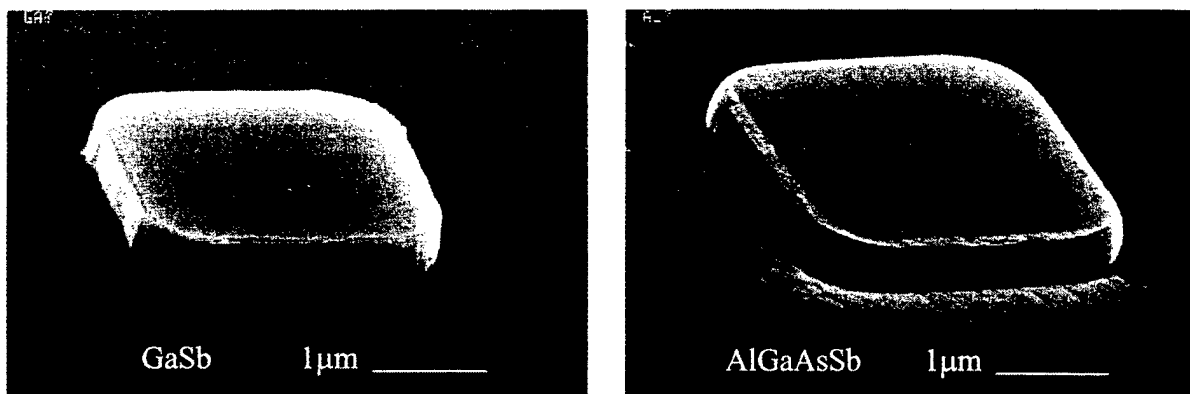


Fig. 3: Scanning electron micrographs of GaSb and  $\text{Al}_{0.9}\text{Ga}_{0.1}\text{As}_{0.07}\text{Sb}_{0.93}$  features etched in  $\text{BCl}_3/\text{Ar}$  plasma. The plasma conditions are: 750 W ICP power, 2 mTorr pressure, 25 sccm  $\text{BCl}_3$  flow rate, 5 sccm Ar flow rate, and 100 V DC self-bias.

Fig. 3 shows scanning electron micrographs of GaSb and AlGaAsSb patterned in a  $\text{BCl}_3/\text{Ar}$  plasma. For a wide range of conditions, the  $\text{BCl}_3/\text{Ar}$  ICP etch yields smooth surfaces, anisotropic profiles, and roughly equal etch rates between GaSb and AlGaAsSb. However, the etch rates are relatively low (typically less than 4000 Å/min) as a result of limited reactive Cl in the plasma. By adding  $\text{Cl}_2$  to a  $\text{BCl}_3/\text{Ar}$  plasma, the concentration of reactive Cl can be significantly enhanced. In Fig. 4, the etch rates and selectivity of AlGaAsSb, GaSb, and InSb are plotted as a function of % $\text{Cl}_2$  in a  $\text{BCl}_3/\text{Cl}_2/\text{Ar}$  plasma. The etch rates of GaSb and AlGaAsSb are enhanced substantially with increasing  $\text{Cl}_2$  percentage in the chemistry, indicating a chemical reaction limited etch regime. Over the entire range of % $\text{Cl}_2$  in the plasma, the etch is non-selective between GaSb and AlGaAsSb. The etch rate of InSb remains low and independent of  $\text{Cl}_2$  concentration due to the low volatility of the  $\text{InCl}_3$  etch product. The GaSb:InSb etch selectivity increases as more  $\text{Cl}_2$  is introduced to the plasma, primarily due to an enhanced GaSb etch rate at higher  $\text{Cl}_2$  concentration.

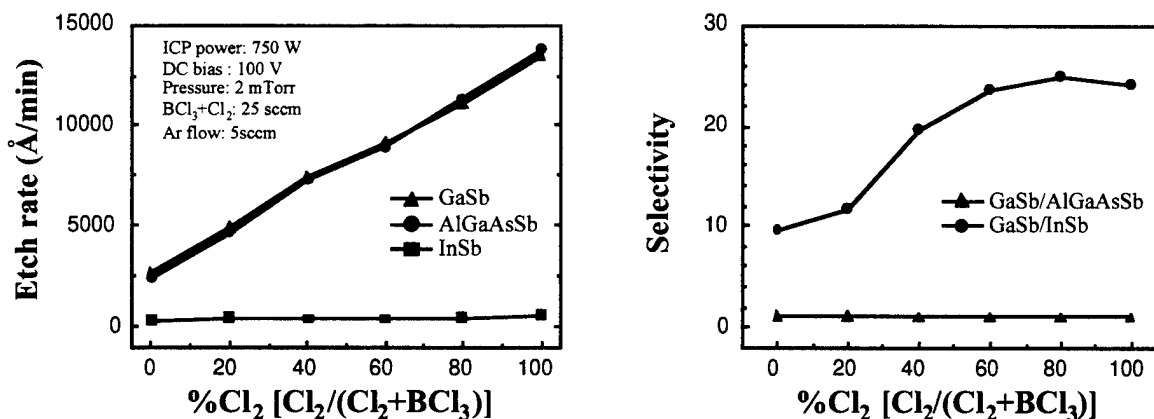


Fig. 4: ICP etch rates and selectivity of GaSb, AlGaAsSb and InSb as a function of % $\text{Cl}_2$  in a  $\text{BCl}_3/\text{Cl}_2/\text{Ar}$  plasma. The plasma conditions are: 750 W ICP power, 100 V DC bias, 2 mTorr pressure, and 5 sccm Ar flow rate. The total flow of  $\text{BCl}_3$  and  $\text{Cl}_2$  is 25 sccm.

Fig. 5 shows the etch rates and selectivity of AlGaAsSb, GaSb, and InSb as a function of DC bias in a  $\text{BCl}_3/\text{Cl}_2/\text{Ar}$  plasma. The plasma conditions are 750 W ICP power, 2 mTorr chamber pressure, 15 sccm  $\text{BCl}_3$ /10 sccm  $\text{Cl}_2$ /5 sccm Ar flow rate. The etch rates of all three materials increase with increasing DC bias. As the DC bias increases from 50 V to 400 V, the etch rates of GaSb and AlGaAsSb increase by a factor of ~2 and the InSb etch rate is enhanced by a factor of ~180. As a result, the GaSb:InSb etch selectivity dropped from ~140 at 50 V DC bias to ~1.6 at 400V. A maximum etch rate of InSb of 8460 Å/min is achieved at 400 V DC bias. The dramatic increase of InSb etch rate suggests that  $\text{InCl}_3$  is removed more efficiently under high-energy ion

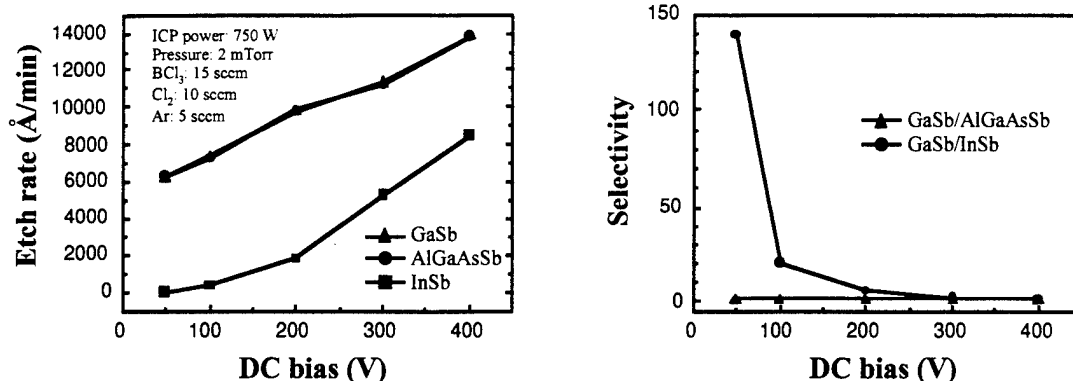


Fig. 5: ICP etch rates and selectivity of GaSb, AlGaAsSb, and InSb as a function of DC bias in a BCl<sub>3</sub>/Cl<sub>2</sub>/Ar plasma. The plasma conditions are: 750 W ICP power, 2 mTorr pressure, 15 sccm BCl<sub>3</sub>/10 Cl<sub>2</sub>/5 sccm Ar flow rate.

bombardment and the etch of InSb in Cl-based plasmas is limited by physical sputtering desorption of the etch product. The GaSb:AlGaAsSb etch selectivity remains ~1.

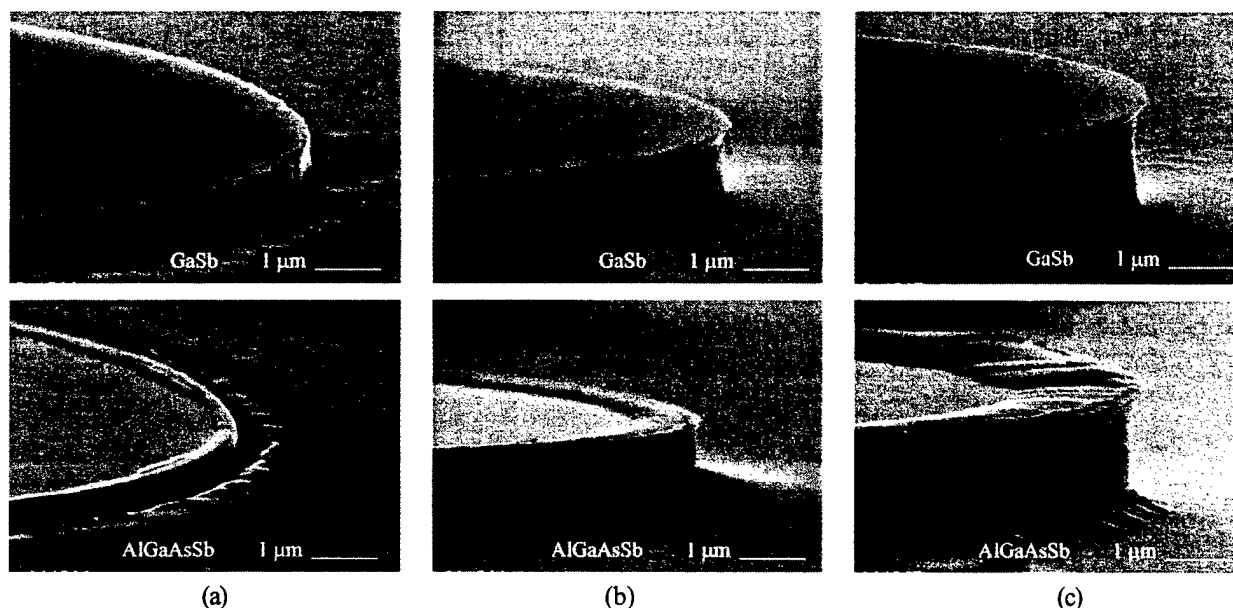


Fig. 6: Scanning electron micrographs of GaSb and AlGaAsSb etched in a BCl<sub>3</sub>/Cl<sub>2</sub>/Ar ICP discharges at (a) 25 sccm BCl<sub>3</sub>/5 sccm Ar; (b) 20 sccm BCl<sub>3</sub>/5 sccm Cl<sub>2</sub>/5 sccm Ar; (c) 15 sccm BCl<sub>3</sub>/10 sccm Cl<sub>2</sub>/5 sccm Ar. The other etch parameters are 750 W ICP power, 100 V DC bias, and 2 mTorr chamber pressure.

Fig. 6 shows SEM micrographs of GaSb and AlGaAsSb samples etched in a BCl<sub>3</sub>/Cl<sub>2</sub>/Ar plasma at different Cl<sub>2</sub> percentage concentrations. For most plasma conditions that are investigated, the BCl<sub>3</sub>/Cl<sub>2</sub>/Ar ICP discharge yields smooth etched surfaces. Vertical and smooth sidewalls are obtained on GaSb and AlGaAsSb samples that are etched in 25 sccm BCl<sub>3</sub>/5 sccm Ar. However, as the Cl<sub>2</sub> concentration increases, a sidewall roughness and undercutting is observed due to the lateral etch. The expanded edge of the etched feature on AlGaAsSb samples is attributed to lateral oxidation of the AlGaAsSb layer through the exposed sidewall. These SEM pictures were taken 4 months after they were etched. The oxidation might occur during the etch because of the existence of O<sub>2</sub> and water vapor in the plasma chamber, or during the time exposed to the air after etch. As the etch depth increases, the expanded edge is elongated in the direction normal to the edge, possibly due to an accelerated oxidation rate.

Fig. 7 shows the SEM micrographs of III-V antimonide samples etched under plasma conditions of 750 W ICP power, 400 V DC bias, 2 mTorr pressure, and 15 sccm BCl<sub>3</sub>/10 sccm Cl<sub>2</sub>/5 sccm Ar flow rate. The etch rates are significantly enhanced because of the more efficient sputtering desorption of etch products under high energy ion bombardment. However, the etch

yields an undercutting profile due to the breakdown of the mask edge. The extreme stress created in the AlGaAsSb epi-layer by oxidation causes its delamination from the substrate.

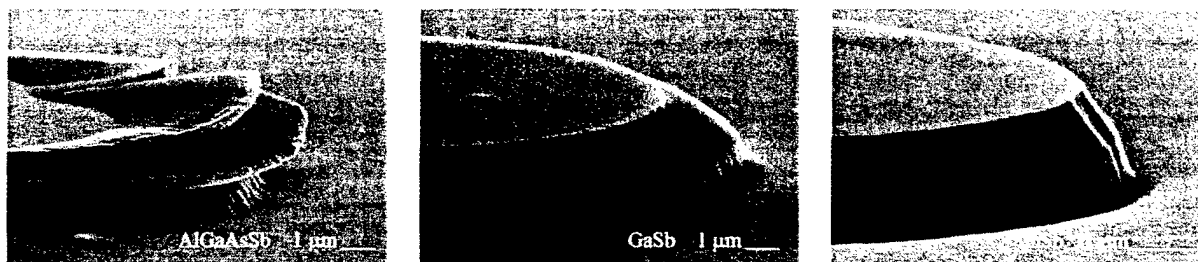


Fig. 7: SEM pictures of GaSb, AlGaAsSb and InSb etched in a  $\text{BCl}_3/\text{Cl}_2/\text{Ar}$  ICP discharges. The plasma conditions are 750 W ICP power, 400 V DC bias, 2 mTorr pressure, and 15 sccm  $\text{BCl}_3$ /10 sccm  $\text{Cl}_2$ /5 sccm Ar flow rate.

## Conclusions

ICP etching characteristics of III-V antimonides have been studied in  $\text{BCl}_3/\text{Ar}$ ,  $\text{Cl}_2/\text{Ar}$ , and  $\text{BCl}_3/\text{Cl}_2/\text{Ar}$  plasmas. Due to a limited reactive Cl supply, the  $\text{BCl}_3/\text{Ar}$  ICP etching mechanism is dominated by ion bombardment and physical desorption of etch products. Smooth, anisotropic, and non-selective etching of GaSb and AlGaAsSb has been achieved in  $\text{BCl}_3/\text{Ar}$  ICP discharges, which is favorable for making dry-etched laser mirrors or deep gratings. By introducing  $\text{Cl}_2$  into a  $\text{BCl}_3/\text{Ar}$  plasma, the concentration of reactive Cl increases substantially, resulting in a significantly enhanced etch rate of III-V antimonides. Etch rates of 1.4  $\mu\text{m}/\text{min}$  for GaSb and AlGaAsSb and 0.85  $\mu\text{m}/\text{min}$  for InSb have been achieved in  $\text{BCl}_3/\text{Cl}_2/\text{Ar}$  ICP discharges. Compared to  $\text{Cl}_2/\text{Ar}$ ,  $\text{BCl}_3/\text{Ar}$  and  $\text{BCl}_3/\text{Cl}_2/\text{Ar}$  ICP etching yields much smoother etched surfaces on AlGaAsSb, indicating that  $\text{BCl}_3$  removes the Al in AlGaAsSb more efficiently, possibly due to improved sputtering or the  $\text{O}_2$  gettering effect of  $\text{BCl}_3$  that reduces surface oxidation.

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# Inductively Coupled Plasma Etching of III-V Antimonides in $\text{BCl}_3/\text{Ar}$ and $\text{Cl}_2/\text{Ar}$

L. Zhang, L. F. Lester,

*Center for High Technology Materials, University of New Mexico, Albuquerque, New Mexico  
87106*

R. J. Shul, C. G. Willison,

*Sandia National Laboratories, Albuquerque, New Mexico 87185*

R. P. Leavitt

*Army Research Laboratory, Adelphi, MD 20783-1145*

## Abstract

Inductively coupled plasma (ICP) etching characteristics of GaSb and AlGaAsSb have been investigated in  $\text{BCl}_3/\text{Ar}$  and  $\text{Cl}_2/\text{Ar}$  plasmas. The etch rates and selectivity between GaSb and AlGaAsSb are reported as functions of plasma chemistry, ICP power, RF self-bias, and chamber pressure. It is found that physical sputtering desorption of the etch products plays a dominant role in  $\text{BCl}_3/\text{Ar}$  ICP etching, while in  $\text{Cl}_2/\text{Ar}$  plasma, the chemical reaction dominates the etching. GaSb etch rates exceeding  $2\text{ }\mu\text{m}/\text{min}$  are achieved in  $\text{Cl}_2/\text{Ar}$  plasmas with smooth surfaces and anisotropic profiles. In  $\text{BCl}_3/\text{Ar}$  plasmas, etch rates of  $5100\text{ }\text{\AA}/\text{min}$  and  $4200\text{ }\text{\AA}/\text{min}$  are obtained for GaSb and AlGaAsSb, respectively. The surfaces of both GaSb and AlGaAsSb etched in  $\text{BCl}_3/\text{Ar}$  plasmas remain smooth and stoichiometric over the entire range of plasma conditions investigated. This result is attributed to effective removal of etch products by physical sputtering. For a wide range of plasma conditions, the selectivity between GaSb and AlGaAsSb is close to unity, which is desirable for fabricating etched mirrors and gratings for Sb-based mid-IR laser diodes.

## Introduction

Semiconductor lasers emitting in the 2-5  $\mu\text{m}$  wavelength range are potential sources for various applications including, laser radar (LADAR)<sup>1</sup>, chemical sensing<sup>2</sup>, and infrared countermeasures (IRCM).<sup>3</sup> The predominant materials in devices operating at these wavelengths are the group-III antimonides, from which broad area laser diodes emitting from 1.6–4.5  $\mu\text{m}$  have been demonstrated.<sup>4–6</sup> Fabrication of more sophisticated device structures requires well-controlled etch techniques to achieve reliable pattern transfer. Compared to wet chemical etching, dry etching provides a more anisotropic profile, greater repeatability, improved etch depth control, and higher aspect ratio. Dry etching is especially important for patterning III-Sb's with high Al concentration because wet etching of these materials yields slow etch rates and rough surfaces.<sup>7</sup> Since wet etching of AlGaAsSb commonly leaves a heavily oxidized surface, only single-step etching is achievable with any predictability. There is surprisingly little in the literature concerning dry etching of III-Sb compounds except the binaries, InSb and GaSb. Pearton, *et. al.*, have investigated electron cyclotron resonance (ECR) etching and reactive ion etching (RIE) of InSb and GaSb using various chemistries.<sup>8–11</sup> GaSb etch rates of 5000 $\text{\AA}/\text{min}$ , 3000 $\text{\AA}/\text{min}$ , and 2500 $\text{\AA}/\text{min}$  were reported in  $\text{Cl}_2/\text{Ar}$ ,  $\text{BCl}_3/\text{Ar}$ , and  $\text{CH}_4/\text{H}_2/\text{Ar}$  ECR discharges, respectively. This paper presents results of inductively coupled plasma (ICP) etching as an attractive alternative to ECR techniques for patterning antimonides.

Compared to ECR sources, it is believed that ICP sources are easier to scale-up, more economical in terms of cost and power requirements, and have more mature automatic tuning technology.<sup>12</sup> ICP plasmas are formed in a dielectric vessel encircled by an inductive coil into which RF power is applied. The alternating electrical field between the coils induces a strong alternating magnetic field that confines electrons and enhances ion density. Anisotropic etch profiles are obtained by superimposing an RF-induced DC self-bias on the sample to control the ion energy independent of the ion density. In this paper, ICP etch results of GaSb and AlGaAsSb

in  $\text{BCl}_3/\text{Ar}$  and  $\text{Cl}_2/\text{Ar}$  plasmas are presented. The etch rate and selectivity are reported as a function of ICP power, DC self-bias, plasma chemistry, and chamber pressure.

## **Experiment**

The samples used in this study are undoped GaSb and undoped  $\text{Al}_{0.9}\text{Ga}_{0.1}\text{As}_{0.07}\text{Sb}_{0.93}$  grown lattice-matched to GaSb by molecular beam epitaxy (MBE). Etching is performed in a load-locked Plasma-Therm SLR 770 ICP reactor with the ICP source operating at 2 MHz. Energetic ion bombardment is provided by superimposing a DC self-bias (13.56 MHz) on the sample. For all experiments, the automatic matching network is set to the DC self-bias control mode that varies the input RF power to match the set point of the DC self bias. Etch gases are introduced through an annular region at the top of the chamber. All samples are mounted using vacuum grease on an anodized Al carrier that is clamped to the cathode and backside-cooled with He gas. The temperature of the electrode is set to 10 °C. Samples are patterned with AZ 4330 photoresist and etched for 2.5 min in the plasma. The etch depth is measured using a Dektak stylus profilometer after the photoresist is removed. Surface morphology, anisotropy, and sidewall undercutting are evaluated with a scanning electron microscope (SEM). Unless otherwise mentioned, the baseline etch parameters are 750 W ICP power, 100 V DC self-bias, 2 mTorr chamber pressure, 25 sccm  $\text{BCl}_3$  or  $\text{Cl}_2$  flow rate, and 5 sccm Ar flow rate.

## **Results**

Fig. 1 shows ICP etch rates for GaSb and AlGaAsSb and etch selectivity between them as a function of DC self-bias in a  $\text{BCl}_3/\text{Ar}$  discharge. Etch rates increase near linearly with increasing DC bias voltage, indicating physical sputtering desorption plays a significant role in  $\text{BCl}_3/\text{Ar}$  ICP etch of these materials. Etch rates of GaSb and AlGaAsSb rise by a factor of 2.5 as DC bias increases from 50 to 300 V. This result is attributed to enhanced ion bombardment and sputter desorption of etch products due to higher ion energies at higher DC bias. The simultaneous

increase in the GaSb and AlGaAsSb etch rates yields a relatively constant etch selectivity between the two materials of approximately  $1.34 \pm 0.12$ .

In Fig. 2, ICP etch rates and the selectivity are plotted as a function of percent composition of  $\text{BCl}_3$  in the chemistry. The total flow rate of  $\text{BCl}_3$  and Ar is held constant at 30 sccm. The data shows a relatively small variation in the GaSb etch rate over the entire range of  $\text{BCl}_3$  composition. This observation suggests that in a  $\text{BCl}_3/\text{Ar}$  ICP mixture, the effect of a higher concentration of reactive Cl on the GaSb etch rate (at a higher  $\text{BCl}_3/\text{Ar}$  ratio) is compensated by the effect of a lower Ar ion sputtering rate. To further verify this assumption, an ICP etch of GaSb and AlGaAsSb in  $\text{BCl}_3/\text{N}_2/\text{Ar}$  is carried out to decouple the physical and chemical component of the etching mechanism. Generally, adding  $\text{N}_2$  to a  $\text{BCl}_3/\text{Ar}$  plasma enhances the dissociation of  $\text{BCl}_3$ , resulting in a higher concentration of reactive Cl and, consequently, a higher etch rate.<sup>1314</sup> Based on a previous study of GaN ICP etching in  $\text{BCl}_3/\text{N}_2$  which shows a peak GaN etch rate at 40%  $\text{N}_2$ ,<sup>15</sup> a chemistry of 15 sccm  $\text{BCl}_3$ /10 sccm  $\text{N}_2$ /5 sccm Ar was chosen to etch GaSb. An etch rate of 5536 Å/min is obtained for GaSb and 4428 Å/min for AlGaAsSb, much higher than the rates obtained in the 25 sccm  $\text{BCl}_3$ /5 sccm Ar ICP discharge shown in Fig. 2. This outcome indicates that the etching is limited by the reactive Cl supply. It also confirms that the physical and chemical component are balanced in the  $\text{BCl}_3/\text{Ar}$  ICP etch, producing a relatively small variation in the GaSb etch rate with changes in  $\text{BCl}_3$  composition. In contrast, the AlGaAsSb ICP etch rate shows a significant increase when  $\text{BCl}_3$  is first introduced into the pure Ar plasma. The etch rate grows by a factor of more than 5 when the  $\text{BCl}_3$  concentration is raised from 0 to 33%, indicating that  $\text{BCl}_3$  removes Al more efficiently than Ar due to the high volatility of  $\text{AlCl}_3$  etch product. A further increase in the  $\text{BCl}_3$  concentration shows little effect on the etch rate of AlGaAsSb. The etch selectivity between GaSb and AlGaAsSb is  $\sim 6:1$  for pure Ar plasma and drops to  $\sim 1.2:1$  as  $\text{BCl}_3$  is introduced into the plasma.

The etch rates and selectivity in  $\text{BCl}_3/\text{Ar}$  as a function of ICP source power are displayed in Fig. 3. The rates rise significantly with increasing ICP power since a larger ICP power creates more chlorine radicals and a higher ion flux, enhancing both the chemical and physical etch

components of the process. The selectivity decreases from 4.2 to 1.3 as the ICP power is increased from 250 W to 500 W, then stays relatively constant for higher powers. The higher selectivity at low ICP power is attributed to a low etch rate of AlGaAsSb due to fewer chlorine radicals.

The dependence of the etching on the process pressure is shown in Fig. 4. An initial etch rate increase as pressure rises from 1 mTorr to 5 mTorr is followed by a decrease at pressures greater than 5 mTorr. The initial increase is attributed to a higher reactive Cl concentration at higher pressure, implying a reactant limited etch regime at low pressure. The decrease in the high pressure range is caused by lower kinetic energy of the ions due to more collisions, suggesting a sputtering desorption limited etch mechanism. The selectivity remains low for the entire pressure range that is studied. Fig. 5 shows scanning electron micrographs of GaSb and AlGaAsSb after patterning in a  $\text{BCl}_3/\text{Ar}$  plasma. For a wide range of conditions, the etch yields smooth surfaces, anisotropic profiles, and roughly equal etch rates between GaSb and AlGaAsSb. This process is most favorable for making dry etched facets for laser mirrors or deep gratings.

In contrast to the  $\text{BCl}_3/\text{Ar}$  ICP etch of III-V antimonides which shows physical sputtering dominated etch characteristics due to a limited reactive Cl supply, the  $\text{Cl}_2/\text{Ar}$  ICP etch mechanism is strongly dominated by chemical reaction. Due to the surface roughness of AlGaAsSb etched in  $\text{Cl}_2/\text{Ar}$  plasmas, an accurate measurement of etch depth on these samples is unachievable; therefore, only GaSb etch rates in  $\text{Cl}_2/\text{Ar}$  plasmas are available. In Fig. 6, the GaSb etch rate is plotted as a function of DC bias. Note that the GaSb etch rate is much higher in a  $\text{Cl}_2/\text{Ar}$  plasma than in a  $\text{BCl}_3/\text{Ar}$  mixture for similar process conditions, suggesting a much higher concentration of reactive Cl in a  $\text{Cl}_2$  plasma as compared to a  $\text{BCl}_3$  plasma. Despite a significant change in DC bias from 50 to 200 V, the rate increases only ~20%. As the DC bias is increased further to 300 V, the etch rate actually decreases slightly. This weak dependence of etch rate on DC bias indicates that the physical sputtering is not a dominant factor in  $\text{Cl}_2/\text{Ar}$  ICP etching. The decrease in etch rate at 300 V may be related to sputtering desorption of reactive species before they have time to react with the III-V antimonides at the surface.

Fig. 7 shows the etch rate as a function of %Cl<sub>2</sub> in a Cl<sub>2</sub>/Ar chemistry. The etch rate increases monotonically with increasing Cl<sub>2</sub> percentage as would be expected from the increased reactive chlorine density in the chamber. Notice that the GaSb etch rate is substantially enhanced (by a factor of > 6) as the Cl<sub>2</sub> percentage increases from 0 to 100%, indicating that the chemical reaction dominates the ICP etching of GaSb in the Cl<sub>2</sub>/Ar chemistry. In Fig. 8, the etch rate of GaSb is plotted as a function of ICP source power. The etch rate increases significantly with increasing ICP power up to 750 W, caused by a higher concentration of reactive species in the plasma and higher ion flux. However, the etch rate remains relatively constant above 750 W, possibly due to saturation of the reactive species at the surface or creation of an adsorption-limited regime. In Fig. 9, the GaSb etch rate is shown as a function of process pressure. At high pressure, the mean free path decreases and the collisional frequency increases. This causes a loss of ion kinetic energy and, typically, lowers the etch rate as a result of a decreased physical sputtering effect. However, the GaSb etch rate in Cl<sub>2</sub>/Ar increases monotonically up to 10 mTorr. This data indicates that the physical sputtering has little effect on the etch rate, that the chemical reaction completely dominates the process, and that the etching is probably reactant-limited.

Fig. 10 shows scanning electron micrographs of GaSb etched in Cl<sub>2</sub>/Ar ICP discharges at different Cl<sub>2</sub> percentage concentrations and pressures. The etched surface remains smooth for all the samples etched at 2 mTorr (a, b, and c). A smooth and vertical sidewall is obtained at 10 sccm Cl<sub>2</sub> and 20 sccm Ar. However, as the Cl<sub>2</sub> concentration increases, sidewall roughness and undercutting is observed due to lateral etching. This effect becomes more obvious in Figure 10 (d), which shows the surface and profile of GaSb etched at 10 mTorr. At high pressure, the sidewall profile is severely undercut and poorly defined because of a lower mean free path, collisional scattering of ions, and increased lateral etching of GaSb. Surface roughness is also observed at 10 mTorr.

## **Conclusions**

ICP etching characteristics of GaSb and AlGaAsSb have been studied in BCl<sub>3</sub>/Ar and Cl<sub>2</sub>/Ar plasmas. The Cl<sub>2</sub>/Ar ICP etching of GaSb exhibits a strong chemically dominated etching

behavior. A GaSb etch rate as high as 4.6  $\mu\text{m}/\text{min}$  is achieved. In  $\text{BCl}_3/\text{Ar}$  plasmas, due to a limited reactive Cl supply, the etching mechanism is dominated by ion bombardment and physical desorption of etch products. Smooth, anisotropic, and non-selective etching of GaSb and AlGaAsSb has been achieved in  $\text{BCl}_3/\text{Ar}$  ICP discharges, which is desirable for making dry-etched facets as laser mirrors. Maximum etch rates of 5100  $\text{\AA}/\text{min}$  and 4200  $\text{\AA}/\text{min}$  are obtained for GaSb and AlGaAsSb, respectively. Compared to  $\text{Cl}_2/\text{Ar}$ ,  $\text{BCl}_3/\text{Ar}$  ICP etching yields much smoother etched surfaces on AlGaAsSb, indicating that  $\text{BCl}_3$  removes the Al in AlGaAsSb more efficiently, possibly due to improved sputtering (higher mass ions) or the  $\text{O}_2$  gettering effect of  $\text{BCl}_3$  which reduces surface oxidation.

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## Figure Captions

Fig. 1. DC bias dependence of the GaSb and  $\text{Al}_{0.9}\text{Ga}_{0.1}\text{As}_{0.07}\text{Sb}_{0.93}$  etch rate and selectivity in a  $\text{BCl}_3/\text{Ar}$  plasma. Unless otherwise noted the plasma conditions are: 750 W ICP power, 2 mTorr pressure, 25 sccm  $\text{BCl}_3$  or  $\text{Cl}_2$  flow rate, 5 sccm Ar flow rate, and 100 V DC self-bias.

Fig. 2. Etch rates and selectivity of GaSb and  $\text{Al}_{0.9}\text{Ga}_{0.1}\text{As}_{0.07}\text{Sb}_{0.93}$  as a function of %  $\text{BCl}_3$  in a  $\text{BCl}_3/\text{Ar}$  plasma. The total gas flow rate is 30 sccm.

Fig. 3. Etch rates and selectivity versus ICP power in a  $\text{BCl}_3/\text{Ar}$  plasma.

Fig. 4. Etch rates and selectivity of GaSb and  $\text{Al}_{0.9}\text{Ga}_{0.1}\text{As}_{0.07}\text{Sb}_{0.93}$  as function of process pressure in a  $\text{BCl}_3/\text{Ar}$  plasma.

Fig. 5. Scanning electron micrographs of GaSb and  $\text{Al}_{0.9}\text{Ga}_{0.1}\text{As}_{0.07}\text{Sb}_{0.93}$  features etched in  $\text{BCl}_3/\text{Ar}$  plasma. The plasma conditions are: 750 W ICP power, 2 mTorr pressure, 25 sccm  $\text{BCl}_3$  flow rate, 5 sccm Ar flow rate, and 100 V DC self-bias.

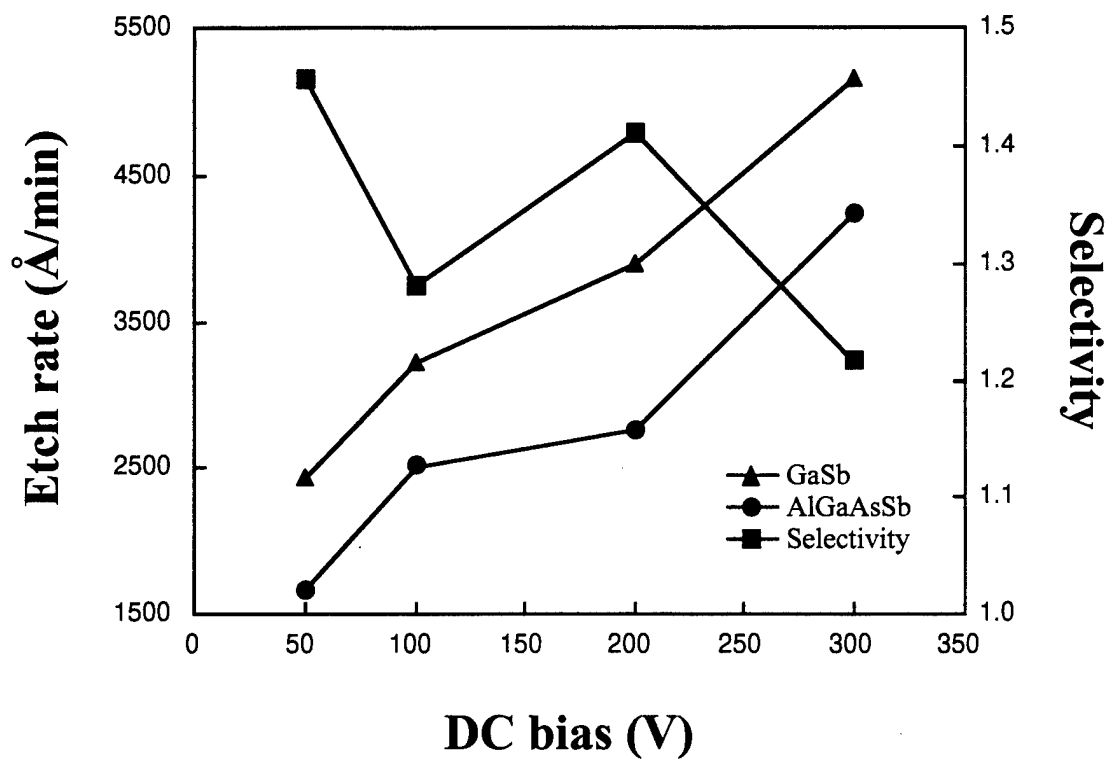
Fig. 6. The etch rate of GaSb in a  $\text{Cl}_2/\text{Ar}$  plasma vs. DC self-bias.

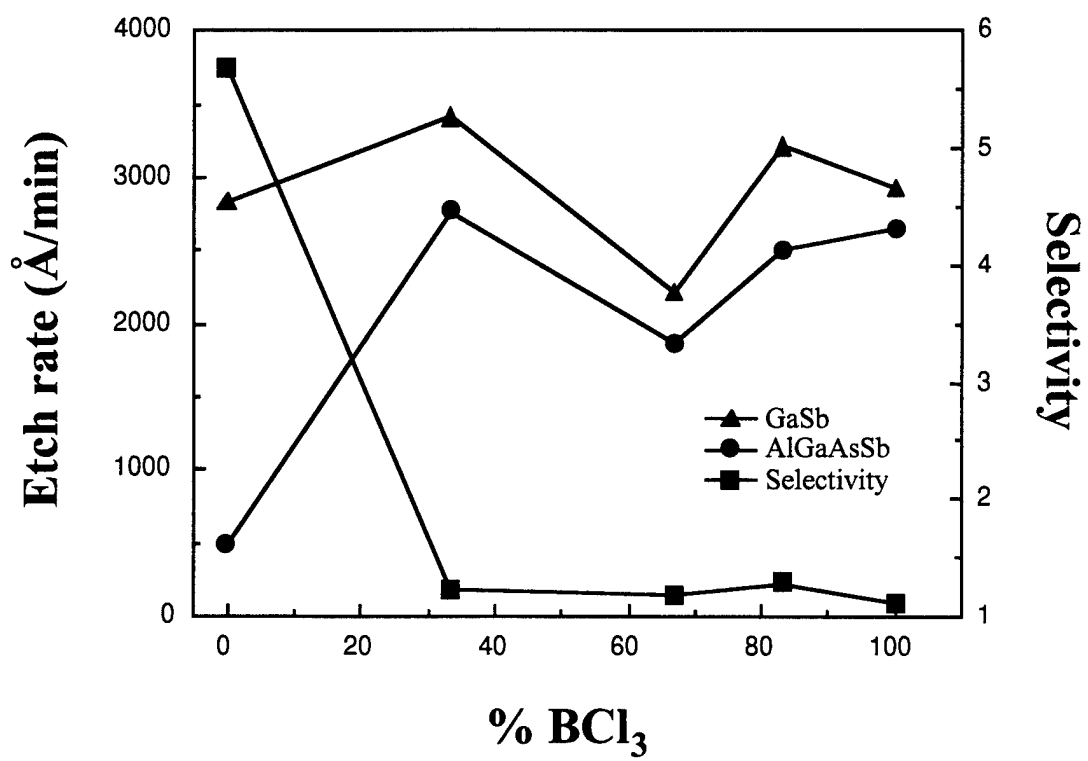
Fig. 7. GaSb etch rate as a function of % $\text{Cl}_2$  in the  $\text{Cl}_2/\text{Ar}$  plasma.

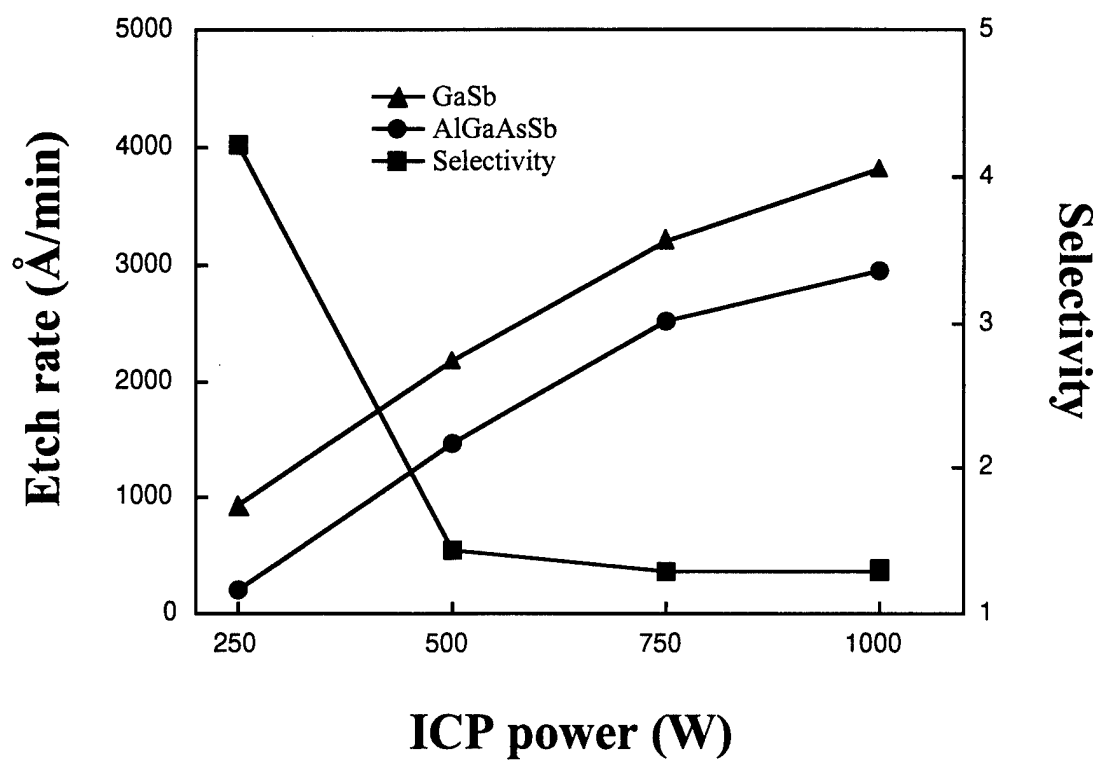
Fig. 8. The GaSb etch rate versus ICP power in a  $\text{Cl}_2/\text{Ar}$  plasma.

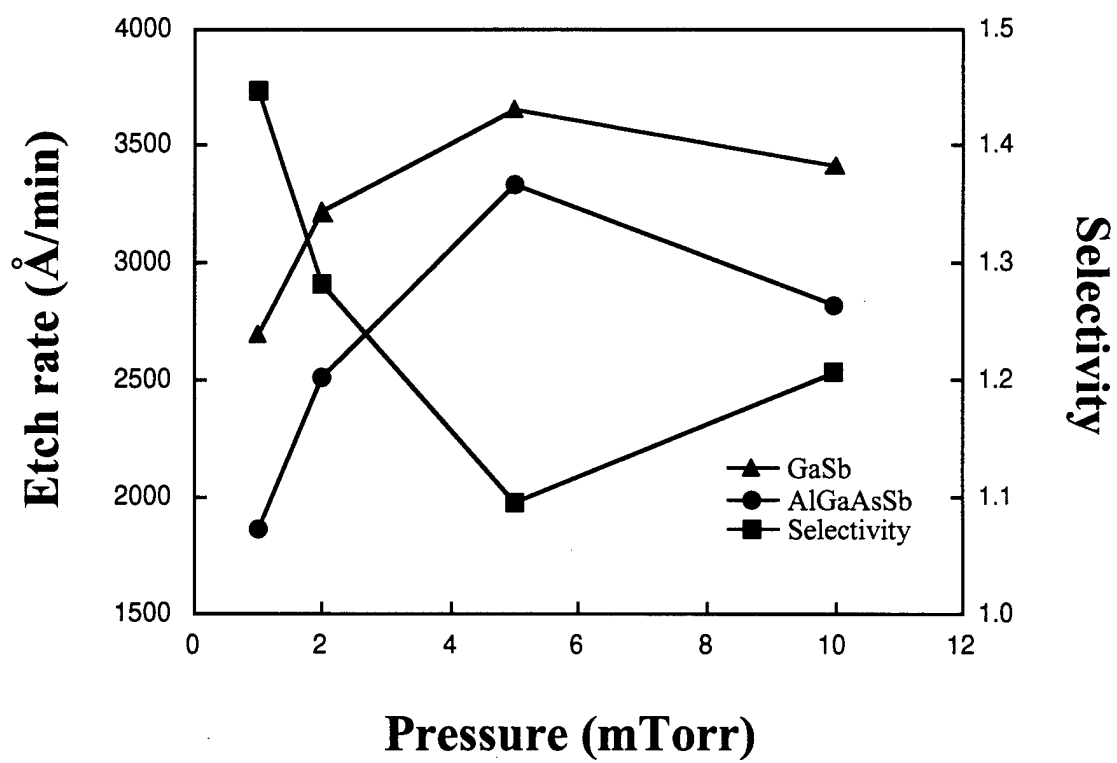
Fig. 9. The process pressure dependence of the GaSb etch rate in a  $\text{Cl}_2/\text{Ar}$  mixture.

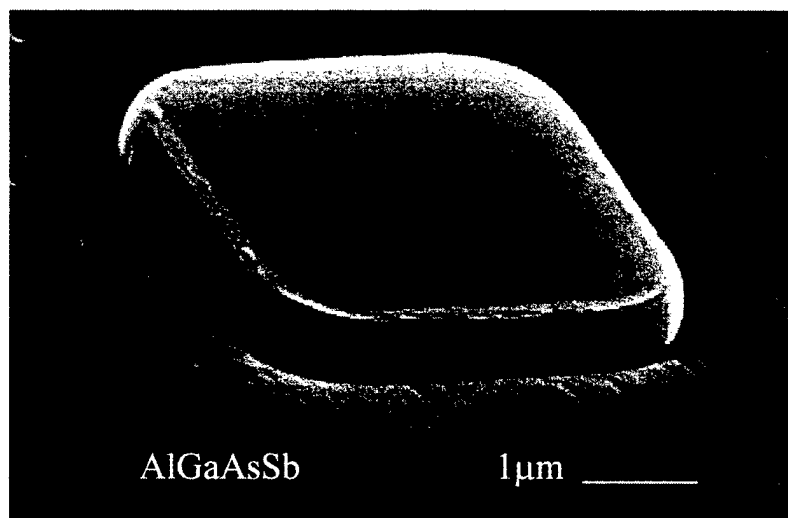
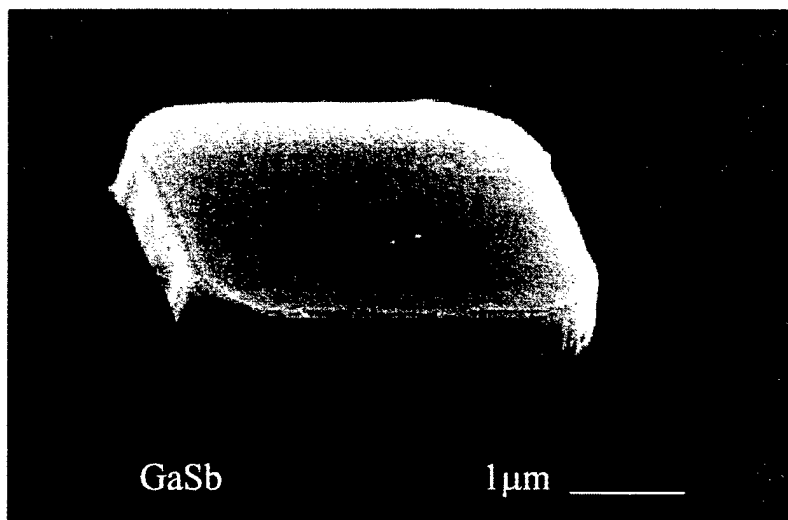
Fig. 10. Scanning electron micrographs of GaSb etched in  $\text{Cl}_2/\text{Ar}$  ICP discharges at (a) 2 mTorr, 10 sccm  $\text{Cl}_2$ /20 sccm Ar; (b) 2 mTorr, 20 sccm  $\text{Cl}_2$ /10 sccm Ar; (c) 2 mTorr, 25 sccm  $\text{Cl}_2$ /5 sccm Ar; (d) 10 mTorr, 25 sccm  $\text{Cl}_2$ /5 sccm Ar. The other etch parameters are 750 W ICP power and 100 V DC bias.

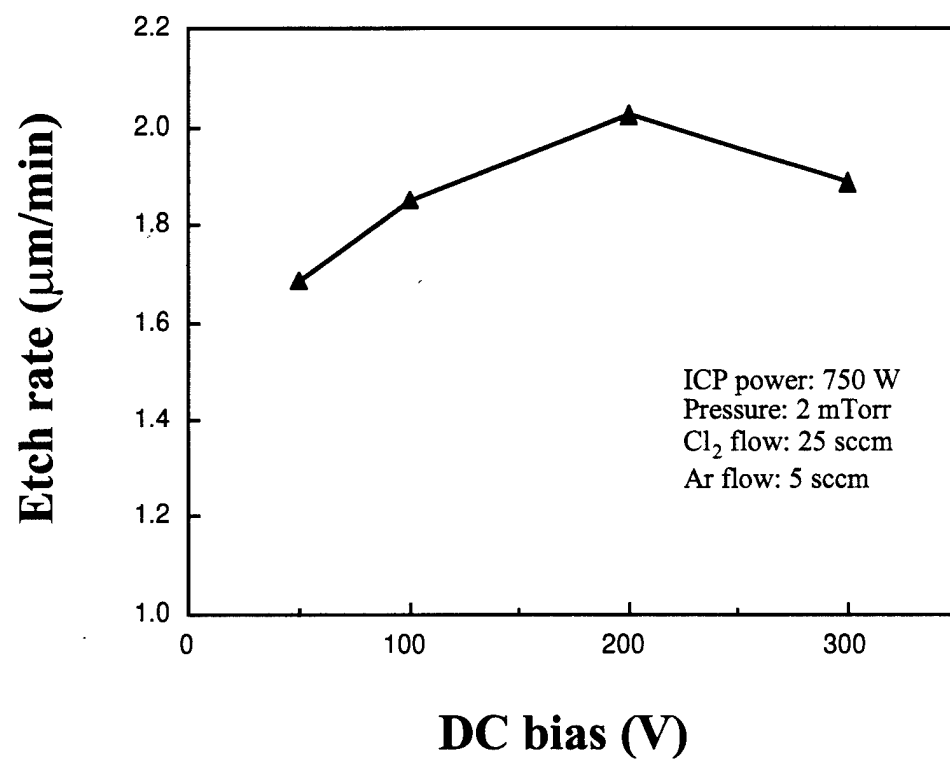




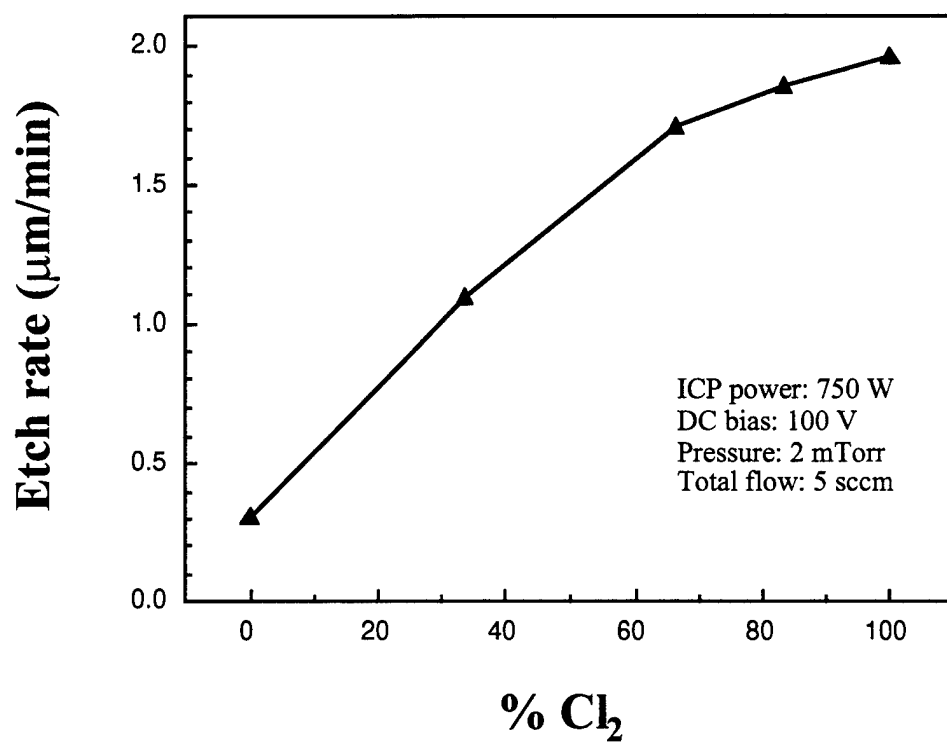


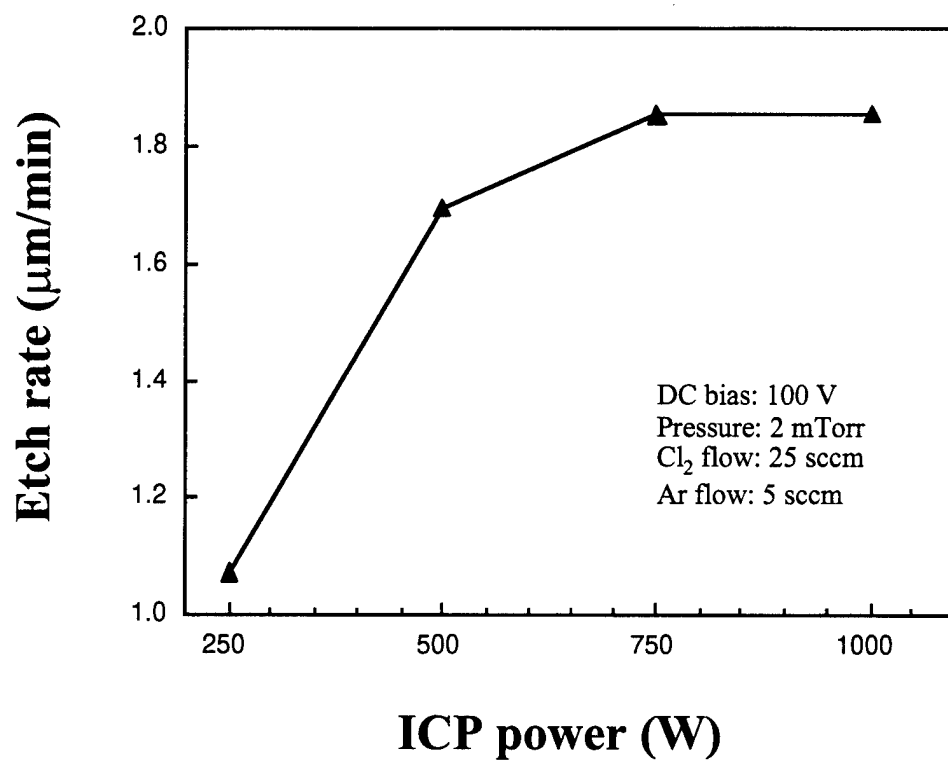


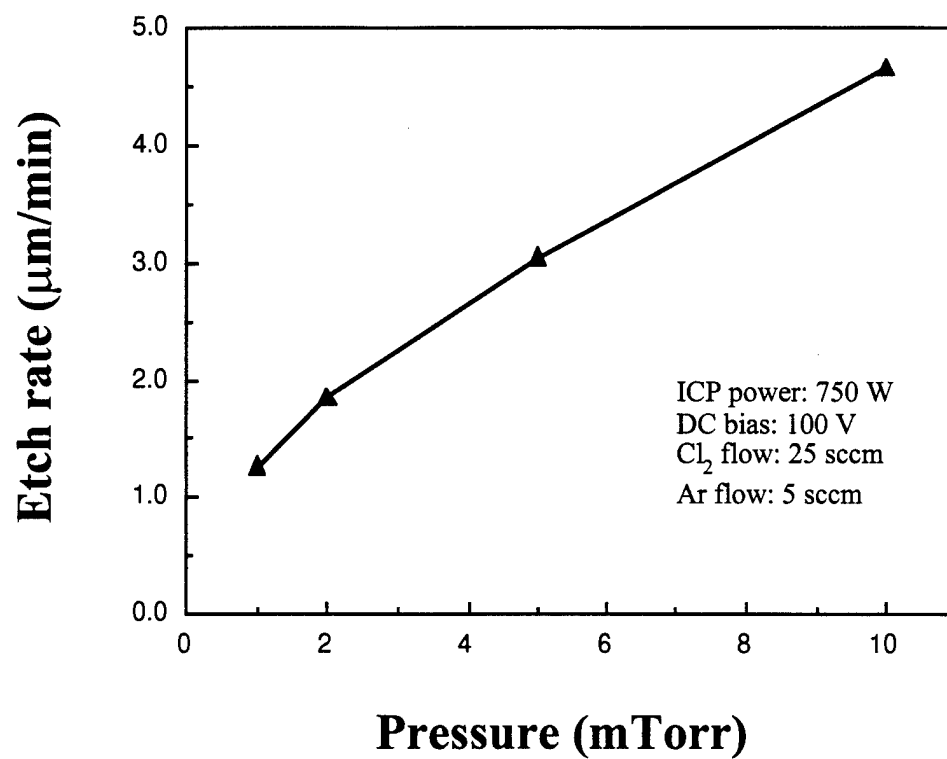


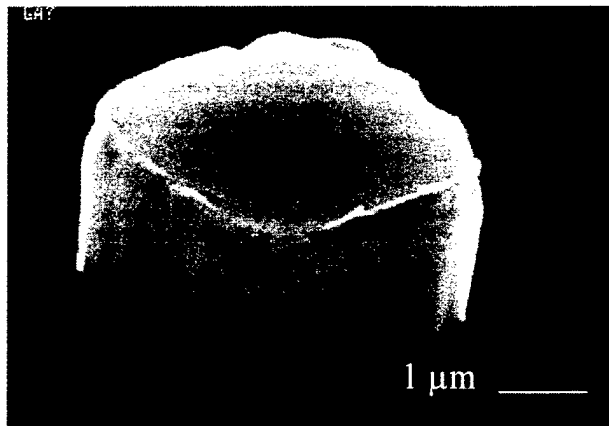




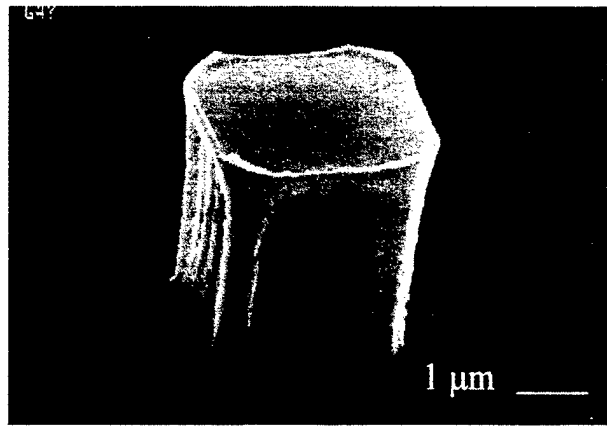




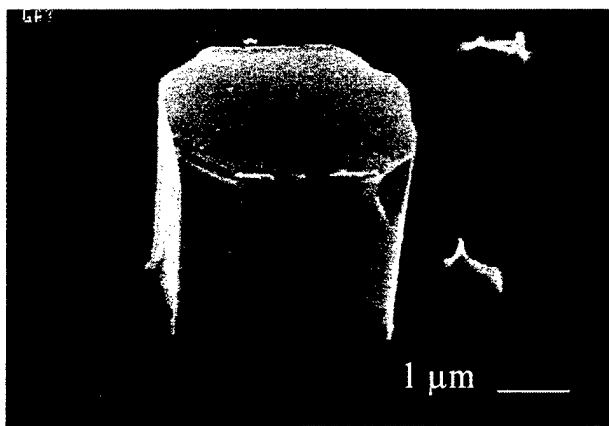




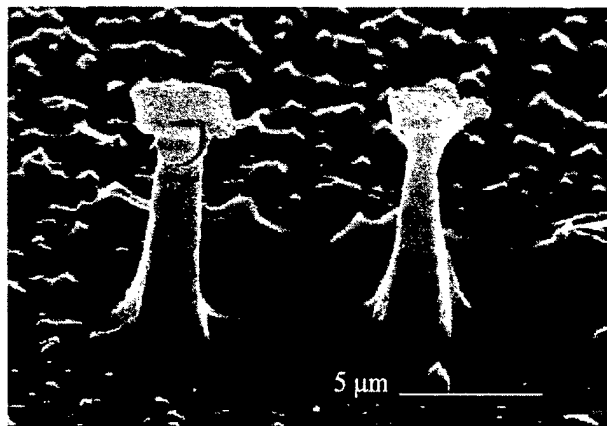
(a)



(c)



(b)



(d)